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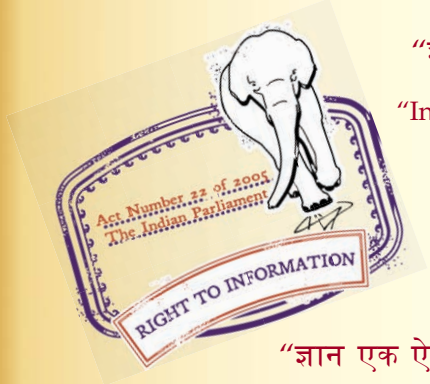
[CHD 25: Soaps and other Surface Active Agents]



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ऐल्किल बेन्जीन सल्फोनिक अम्ल (अम्लीय घोल) — विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

ALKYL BENZENE SULPHONIC ACID (ACID SLURRY) — SPECIFICATION

(First Revision)

UDC 661·715·7

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 1994

Price Group 5

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Soaps and Other Surface Active Agents Sectional Committee had been approved by the Chemical Division Council.

Alkyl benzene which is the starting material for the manufacture of alkyl benzene sulphonic acid, also known as acid slurry in trade, is the single largest raw material used for the production of synthetic detergents throughout the world. Synthetic detergents based on alkyl benzene find extensive application as household and industrial washing and cleaning materials either alone or in combination with other anionic or non-ionic surface active agents.

The molecular mass of alkyl benzene varies from 236 to 256 depending on the source of supply. Alkyl benzene is sulphonated using sulphuric acid, oleum or sulphur trioxide (SO_3) gas. Alkyl benzene sulphonic acid manufactured by these different processes varies in active matter, free acid and also in colour and viscosity.

Alkyl benzene is of two types, branched and linear. The biodegradability of linear alkyl benzene is good while that of branched alkyl benzene is poor. In India, only linear alkyl benzene being used although there is no legislation to ban the use of branched alkyl benzene. The production of linear alkyl benzene in India started in the year 1979. Now the country is self sufficient in linear alkyl benzene.

This standard was first published in 1977.

The technical committee responsible in the formulation of this standard decided to revise it. In this revision, the requirement for packing has been modified besides the amendment No. 1 published in July 1988 has been incorporated. Also, in the method of determination of alkyl benzene sulphonic acid, the molecular mass of alkyl benzene has modified to 235 to 245.

Acid slurry is an important intermediate raw material for manufacture of household and industrial synthetic detergent powders, tablets/cakes and liquids. The limitations of soaps for use in hard water areas led to the rapid development of synthetic detergent industry. The shortage of oils and fats, the main raw materials for production of soaps, and increase in their demand for edible purposes have further helped the growth of synthetic detergent industry in India. At present it is being produced by a large number of units in small scale as well as large scale sector.

The formulation of this standard was taken up on a specific request from Development Commissioner, Small Scale Industries, Government of India with a view to regulating the quality of acid slurry produced in the country.

The composition of the Technical Committee and Subcommittee responsible for the formulation of this Indian Standard is given in Annex G.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ALKYL BENZENE SULPHONIC ACID (ACID SLURRY) — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes requirements and methods of sampling and test for alkyl benzene sulphonic acid (acid slurry).

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard.

IS No.	*Title*
286 : 1978	Methods of sampling and test for soaps (*first revision*)
321 : 1964	Specification for obsolute alcohol (*revised*)
336 : 1976	Specification for ether (*second revision*)
1070 : 1992	Specification for reagent grade water (*third revision*)
2263 : 1979	Methods of preparation of indicator solutions for volumetric analysis (*first revision*)
7597 : 1974	Glossary of terms relating to surface active agents

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 7597 : 1974 shall apply.

4 TYPES

The material shall be of two types depending on active matter content, namely:

a) Type 1, and

b) Type 2.

5 REQUIREMENTS

5.1 Description

The material shall be in the form of a homogeneous viscous liquid, easily pourable and shall not separate into two layers. When neutralized with alkali, it shall form a paste ranging from white to cream in colour.

5.2 The material shall also comply with the requirements prescribed in Table 1.

6 PACKING AND MARKING

6.1 Packing

The material shall be packed in polyethylene containers of suitable size, or any other suitable material or as agreed to between the purchaser and the supplier.

NOTE — If supplied in mild steel, there is likelihood of material getting contaminated with iron particles.

6.2 Marking

The packages shall be securely closed and marked with the following particulars:

a) Name and type of the material;

b) Indication of source of manufacture;

c) Net mass of the material;

d) Batch No. or lot No. in code or otherwise;

e) Month and year of manufacture; and

f) USE HAND GLOVES WHILE HANDLING.

7 SAMPLING

7.1 General

The general precautions, scale of sampling and preparation of test samples, shall be as prescribed in **3.1, 3.2** and **3.3** as applicable for liquid soap respectively of IS 286 : 1978.

7.2 Number of Tests

7.2.1 Tests for determination of characteristics given at Sl No. (i) and (ii) in Table 1 shall be conducted on each of the individual samples separately.

7.2.2 Tests for determination of all the remaining characteristics shall be conducted on the composite sample.

Table 1 Requirements for Alkyl Benzene Sulphonic Acid (Acid Slurry)

(*Clauses* 5.2 *and* 8.1)

Sl No.	Characteristic	Requirement for		Method of Test (Ref to Annex)
		Type 1	Type 2	
(1)	(2)	(3)	(4)	(5)
i)	Active matter as alkyl benzene sulphonic acid, percent by mass, *Min*	95·0	85·0	A & B
ii)	Free alkyl benzene, percent by mass, *Max*	2·0	2·0	C
iii)	Free sulphuric acid, percent by mass, *Max*	2·0	9·0	D
iv)	Colour			
	a) of 10 percent alcohol c solution (mass by mass) in 1/4 in cell on Lovibond Tintometer, not darker than	5 Y + 1 R	3 Y + 0·5 R	E
	b) of solution in distilled water containing 5 percent active matter on Klett colorimeter, *Max* (in Klett)	950	700	F

NOTES

1 Any of the three methods may be followed for routine testing, however, in case of dispute the Lovibond Tintometer method shall be the referee method.

2 For the purpose of calculation, the molecular mass shall be taken as 240.

7.3 Criteria for Conformity

7.3.1 *For Individual Samples*

For each of the characteristics which has been determined on the individual samples (**7.2.1**) the mean (X) and the range (R) of the test results shall be calculated as follows:

$$\text{Mean } (X) = \frac{\text{The sum of test results}}{\text{Number of test results}}$$

Range (R) = The difference between the maximum and the minimum value of the test results.

7.3.1.1 The lot shall be deemed as conforming to the requirement if the expression ($X - 0{\cdot}6\,R$) is greater than or equal to maximum value given in Table 1, and ($X + 0{\cdot}6\,R$) is less than or equal to maximum value given in Table 1.

7.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of other characteristics determined on the composite sample, the test results for each of the characteristics shall satisfy the relevant requirement.

8 TESTS

8.1 Tests to evaluate characteristics prescribed in Table 1 shall be conducted as prescribed in Annex A to E. Reference to relevant clause of Annex B is given in A-01.

8.2 Quality of Reagents

Unless specified othewise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain inpurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF ALKYL BENZENE SULPHONIC ACID

A-0.1 GENERAL

In the method prescribed, the molecular mass of active matter shall be taken based on the molecular mass of alkyl benzene as declared by the supplier. This method shall be used for routine analysis. For more accurate results, molecular mass of sodium alkyl benzene sulphonic acid shall be determined as prescribed in Annex B and then used in calculating the active matter content by this method.

A-1 OUTLINE OF THE METHOD

A-1.1 A solution of the anionic detergent containing added methylene blue is shaken with chloroform, which dissolves the methylene blue salt of the detergent. The mixture is titrated with a cationic-active agent which, after it has combined with all the free anionic detergent begins to displace methylene blue from the salt. The end point is taken when sufficient methylene blue has been displaced into the aqueous layer to produce phases of equal colour intensity. As the reaction is not stoichiometric, it is essential to carry out standardization using a known anionic detergent similar in nature to the unknown.

NOTE — Hypochlorites and sulphites interfere with the detection of the end point and should be eliminated by the addition of ferrous sulphate or hydrozen peroxide respectively.

A-2 APPARATUS

A-2.1 Volumetric Flasks — 1 000, 500 and 250 ml.

A-2.2 Stoppered Graduated Cylinder — 50 ml.

A-2.3 Graduated Cylinder — 50 ml.

A-2.4 Burette — 25 ml.

A-2.5 Pipette — 10 ml.

A-2.6 Beakers — 250 ml.

A-3 REAGENTS

A-3.1 Chloroform — chemically pure.

A-3.2 Sulpuric Acid 5 N Solution

Carefully add 134 ml of sulphuric acid (relative density 1·84) to 300 ml of water and dilute to 1 litre.

A-3.3 Standard Sulphuric Acid — 1·0 N.

A-3.4 Standard Sodium Hydroxide Solution — 1·0 N.

A-3.5 Standard Sodium Lauryl Sulphate Solution — 0·004 M.

Check the purity of the sodium lauryl sulphate as given in **A-3.5.1** and simultaneously prepare the standard solution.

A-3.5.1 *Determination of Purity of Sodium Lauryl Sulphate*

Weigh to the nearest 1 mg, $5 \pm 0{\cdot}2$ g of the material into a 250-ml round bottom flask with ground-glass neck. Add exactly 25 ml of standard sulphuric acid solution (1·0 N) and reflux under a water condenser. During the first 5 to 10 minutes, the solution will thicken and tend to foam strongly; control this by removing the source of heat and swirling the contents of the flask. In order to avoid excessive foaming, instead of refluxing, the solution may be left on a boiling water-bath for one hour. After a further 10 minutes the solution clarifies and foaming ceases. Reflux for a further 1 hour. Remove the source of heat, cool the flask and carefully rinse the condenser with 30 ml of ethanol followed by water. Add a few drops of the phenolphthalein solution and titrate with standard sodium hydroxide solution. Carry out a blank test by titrating 25 ml of sulphuric acid solution (1·0 N) with standard sodium hydroxide solution.

$$\text{The purity of the sodium lauryl sulphate} = \frac{28{\cdot}84\,(V_1 - V_0)\,N_1}{M_1}$$

where

V_0 = volume in ml of standard sodium hydroxide solution used for the blank;

V_1 = volume in ml of standard sodium hydroxide solution used for the sample;

N_1 = Normality of standard sodium hydroxide solution; and

M_1 = mass in g of sodium lauryl sulphate under test.

A-3.5.2 *Procedure*

Weigh to the nearest 1 mg, between 1·14 and 1·16 g of sodium lauryl sulphate and dissolve in 200 ml of water. Transfer to a ground glass stoppered 1 litre one-mark volumetric flask and dilute to the mark with water. Calculate the

molarity, T_1, of the solution by means of the formula:

$$M_1 = \frac{M_2 \times \text{purity, percent by mass}}{288{\cdot}4 \times 100}$$

where

M_2 = mass in g of sodium lauryl sulphate taken.

A-3.6 Standard Benzethonium Chloride Solution* — 0·004 M.

Weigh to the nearest 1 mg between 1·75 and 1·85 g of benzethonium chloride and dissolve in water. Transfer to a ground glass stoppered 1 litre one-mark volumetric flask and dilute to the mark with water.

NOTES

1 In order to prepare a 0·004 M solution, dry the benzethonium chloride at 105°C, weigh 1·792 g to the nearest 1 mg, dissolve in water and dilute to 1 litre. While drying, take care not to raise the temperature beyond 105°C.

2 Other cationic reagents, such as cetyl trimethyl ammonium bromide and benzalkonium chloride, give results identical to those obtained using benzethonium chloride. However these tests have not been carried out in sufficient number to make it possible to state that the result will be identical no matter what the product analysed; for that reason, if benzethonium chloride is not available it is permitted to use another reagent provided that this is stated in the test report. However in case of doubt and always in case of a dispute, only benzethonium chloride should be used.

A-3.7 Phenolphthalein Indicator Solution

Dissolve 1 g of phenolphathalein in 100 ml of 95 percent (*v/v*) ethanol.

A-3.8 Methylene Blue Solution (0·005 Percent)

Dissolve 0·05 g of methylene blue, 50 g of sodium sulphate and 6·8 ml of concentrated sulphuric acid in water and make up the volume to 1 litre with water.

A-3.8.1 Mixed indicators are also available and may be used.

A-3.9 Sample Solution

Weigh a suitable quantity of the sample containing 100 to 160 mg of anionic active matter per 100 ml of solution. For this take an appropriate quantity of sample and neutralize with required quantity of sodium hydroxide solution to get sodium alkyl benzene sulphonate. About 1 g of sodium alkyl benzene sulphonate per 500 ml of the solution is suitable.

* The full name of this chemical is benzyl dimethyl 2-[2-p(1,1,3,3-tetramethylbutyl) phenoxy-ethoxy]-ethyl ammonium chloridemono-hydrate. [$CH_3.CCH_3.C(CH_3)_2\ C_6H_4.OCH_2CH_2OCH_2.\ CH_2N(CH_3)_2\ CHD_2\ C_8H_5$] + $Cl^-.H_2O$]. It is commercially sold under the name 'Hyamine 1622'.

A-4 PROCEDURE

A-4.1 Standardization of Benzethonium Chloride Solution

A-4.1.1 Pipette 10 ml of standard sodium lauryl sulphate solution (*see* **A-3.5**) into a 100-ml graduated cylinder provided with a glass stopper. Add 15 ml of chloroform and 25 ml of methylene blue reagent to the cylinder. Shake well. The chloroform layer (lower) shall be coloured blue or greenish blue.

A-4.1.2 Add from the burette benzethonium chloride solution slowly, initially in portions of 0·2 ml. After each addition, stopper the cylinder, shake well and allow the phases to separate. Initially the chloroform phase will be coloured blue or greenish blue. Towards the end the colour would start migrating to the aqueous layer. Note the reading at which the colour intensity in both the phases is the same when viewed under standard conditions of light, for example, against a white porcelain tile under normal daylight.

A-4.1.3 Calculate the molarity of benzethonium chloride solution as follows:

$$\text{Molarity of benzethonium chloride solution, } T_2 = \frac{10\ T_1}{V_1}$$

where

T_1 = molarity of sodium lauryl sulphate solution, and

V_1 = Volume in ml of benzethonium chloride solution added.

A-4.2 Determination of Anionic Active Matter

Proceed as described in **A-4.1**, taking 10 ml of the sample solution (**A-3.9**) instead of sodium lauryl sulphate solution.

A-4.3 Calculation

Calculate the anionic active matter as alkyl benzene sulphonic acid as follows:

$$\text{Anionic active matter, percent by mass} = \frac{M \times V_2 \times T_2 \times 5}{M_2}$$

where

V_2 = volume in ml of benzethonim chloride solution added;

T_2 = molarity of benzethonium chloride solution (*see* **A-4.1.3**);

M_2 = mass in g of the sample taken; and

M = molecular mass of alkyl benzene sulphonic acid taken for calculation.

ANNEX B

(*Clause* A-0.1)

DETERMINATION OF MOLECULAR MASS OF SULPHONIC ACID OR SODIUM SALT OF SULPHONIC ACID

B-1 APPARATUS

B-1.1 Beakers — 150 and 1 000 ml capacity.

B-1.2 Buchner Flask — 500 ml capacity, fitted with a sintered glass filter funnel (prosity 4).

B-1.3 Evaporating Basin

B-1.4 Separating Funnels — 1 000 ml capacity.

B-1.5 Steam-Bath

B-1.6 Wide-Mouthed Flat Bottomed Flask — 200 ml capacity.

B-1.7 Air-Oven — Preferably electrically heated with temperature control device.

B-2 REAGENTS

B-2.1 Caustic Soda Solution — 10 percent (*m/v*).

B-2.2 Ethyl Alcohol — 30 percent, 96 percent, (*v/v*) and absolute.

B-2.3 Diethyl Ether

B-2.4 Acetone

B-2.5 Phenolphthalein Indicator — *See* IS 2263 : 1979

B-2.6 Methyl Orange Indicator — 0·1 percent (*m/v*).

B-2.7 Ferric Ammonium Sulphate Indicator — Saturate solution.

B-2.8 Standard Sulphuric Acid — Approximately 0·1 N.

B-2.9 Standard Ammonium Thiocyanate Solution — Approximately 0·1 N.

B-2.10 Nitric Acid — Concentrated, relative density 1·42.

B-2.11 Nitro-Benzene

B-2.12 Standard Silver Nitrate Solution — Approximately 0·1 N.

B-3 PROCEDURE

B-3.1 Weigh about 2 g of the material into a 150-ml beaker. Dissolve in minimum quantity of water and neutralize with caustic soda solution. Evaporate on a steam-bath to almost complete dryness. Digest with 50 ml of 96 percent ethyl alcohol by heating on a steam-bath for about 2 minutes. Stir and break up any hard lump with a glass rod flattened at one end. Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel fitted to a Buchner flask to which suction is applied. Repeat the alcoholic digestion in a similar manner with 5 further consecutive 30 ml portions of boiling ethyl alcohol. Filter each extract in turn through the same sintered glass funnel and, finally, wash the residue several times with hot ethyl alcohol to remove all the alcohol solubles. Evaporate the combined filtrate to a small bulk in an evaporating dish and transfer it to a separating funnel. Rinse the evaporating dish once with 50 ml of 96 percent ethyl alcohol and then four times with 50 ml portions of water. Add each wash in turn to the separating funnel. Add 150 ml of diethyl ether. Swirl gently to ensure adequate mixing, and allow the two phases to separate. Run off the aqueous alcoholic layer into a second separating funnel, and extract twice with 75 ml portions of diethyl ether. Transfer the aqueous alcoholic phase into a beaker, and combine the three ether extracts.

B-3.2 Take the combined ether extracts in a clean separating funnel. Wash three times with successive 50 ml portions of 30 percent ethyl alcohol and then with successive 50 ml portions of water until the ether phase is free from alcohol; usually 7 to 10 water-washes are necessary. Combine all the alcoholic and aqueous extracts, neutralize to phenolphthalein and evaporate on a steam-bath until the volume is reduced to about 25 ml. Add an equal volume of absolute alcohol and evaporate to dryness. The solution shall remain just pink to phenolphthalein throughout evaporation. To ensure that the residue is completely anhydrous, add 30 ml of absolute alcohol and again evaporate to dryness. Extract the residue with 30 ml of hot 96 percent ethyl alcohol, stirring and breaking up the solid matter in the dish with a glass rod. Allow the solid matter to settle and decant the hot alcoholic solution through a sintered glass filter funnel fitted to a Buchner flask to which suction is applied. Extract the residue in the dish with six further consecutive 30 ml portions of hot 96 percent ethyl alcohol. Pass each extract in turn through the sintered glass filter. Finally, wash the residue in the sintered glass filter three times with about 20 of hot 96 percent ethyl alcohol from the jet of a wash bottle.

B-3.3 Transfer the filtrate and washings in the Buchner flask to a wide mouthed flat-bottomed flask, evaporate nearly to dryness in a water-bath, and drive off the remaining solvent by directing a gentle stream of dry air into the flask whilst continuously rotating the latter on the water-bath. A thin film of active matter, easy to dry, is thereby obtained. Add 10 ml of acetone, evaporate and remove the last traces of solvent as described above, cool in a desiccator and weigh. Heat the flask for not more than five minutes in an air-oven at a temperature of 100 ± 1°C, gently blow out with a current of air, cool and reweigh. Repeat this drying process until the difference between two successive weighings does not exceed 3 mg.

B-3.4 The extract obtained contains the active matter, some sodium chloride and possibly traces of alkali carbonates which may have passed through the filter in the presence of detergent. Find the percentage of sodium carbonate and sodium chloride in the extract by using a portion of the extract as prescribed in **B-3.4.1** and **B-3.4.2** respectively.

B-3.4.1 *Determination of Alkali Carbonates*

Weigh accurately about 1 g of the extract. Dissolve it in cold water, add a few drops of methyl orange indicator solution and titrate with standard sulphuric acid to methyl orange end point.

B-3.4.1.1 *Calculation*

$$\text{Mass of sodium carbonate, g} = 0{\cdot}053\ V_1 N_1 \times \frac{M_1}{M_2}$$

where

V_1 = volume in ml of standard sulphuric acid solution used,

N_1 = normality of the standard sulphuric acid solution;

M_1 = mass in g of the total extract; and

M_2 = mass in g of the extract taken for analysis.

B-3.4.1.2 Reserve the solution for estimation of chlorides.

B-3.4.2 *Determination of Chlorides*

To the solution remaining after the estimation of alkali carbonates (*see* **B-3.4.1.2**), add 2 ml of concentrated nitric acid and 20 ml of standard silver nitrate solution. Add 3 ml of nitrobenezene and shake vigorously. Titrate with standard ammonium thiocyanate solution using ferric ammonium sulphate as indicator.

B-3.4.2.1 *Calculation*

$$\text{Mass of sodium chloride, g} = 0{\cdot}058\ 5\ (20N_3 - V_2N_2) \times \frac{M_1}{M_2}$$

where

V_2 = volume in ml of standard ammonium thiocynate solution used;

N_2 = normality of standard ammonium thiocynate solution;

N_3 = normality of standard silver nitrate solution;

M_1 = mass in g of the total extract (*see* **B-3.3**); and

M_2 = mass in g of the extract taken for analysis in **B-3.4.1.**

B-3.5 Weigh accurately about 1 g of the extract. Dissolve in water and make up to 500 ml. Follow the titration procedure given in **A-4.2** taking 10 ml of the solution for titration.

B-4 CALCULATION

B-4.1 Molecular mass

$$\text{of alkyl benzene sulphonic acid} = \frac{(100 - M_1 - M_2) \times M}{5 \times V_1 \times T_1} - 22$$

where

M = mass in g of the extract taken;

M_1 = percent by mass of sodium carbonate (*see* **B-3.4**),

M_2 = percent by mass of sodium chloride (*see* **B-3.4.2**).

V_1 = volume in ml of benzethonium chloride solution added;

T_1 = molarity of benzethonium chloride solution; and

22 = factor for converting molecular mass of sulphonate into that of acid slurry.

ANNEX C

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF FREE ALKYL BENZENE

C-1 REAGENTS

C-1.1 Absolute Alcohol — *See* IS 321 : 1964.

C-1.2 Dilute Ethyl Alcohol — 50 percent.

C-1.3 Petroleum Ether — 60 to 80°C boiling range.

C-1.4 Phenolphthalein Solution — *See* IS 2263 : 1979.

C-1.5 Sodium Hydroxide Solution — 2N

C-2 PROCEDURE

C-2.1 Weigh accurately about 5 g of sample (linear alkyl benzene sulphonate) into a 250-ml beaker. Dissolve in 50 ml of alcohol. Neutralize to phenolphthalein with sodium hydroxide solution. Add 50 ml of water. Transfer to a 250-ml separating funnel. Extract twice with 75 ml and 50 ml of petroleum ether respectively. Wash the extract with 50 ml of 50 percent alcohol.

C-2.2 Filter the ether layer through anhydrous sodium sulphate taken on Whatman filter paper No. 1 or equivalent, in a previously weighed 150 ml beaker. Flash off the ether and dry the residue to constant mass.

C-3 CALCULATION

C-3.1 Calculate the free alkyl benzene as follows:

$$\text{Alkyle benzene, percent by mass} = \frac{M_1 \times 100}{M_2}$$

where

M_1 = mass in g of residue; and

M_2 = mass in g of the sample taken for the test.

ANNEX D

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF FREE SULPHURIC ACID

D-1 REAGENTS

D-1.1 Absolute Alcohol — *See* IS 321 : 1964.

D-1.2 Phenolphthalein Solution — *See* IS 2263 : 1979.

D-1.3 Sodium Hydroxide Solution — 40 percent (*m/v*).

D-1.4 Dilute Hydrochloric Acid — 50 percent (*v/v*).

D-1.5 *p*-Toluidine Hydrochloride Solution

Make a solution of 100 g of *p*-toluidine and 78 ml of concentrated hydrochloric acid to one litre in water.

D-1.6 Ethyl Ether — *See* IS 336 : 1964.

D-1.7 Barium Chloride Solution — 10 percent (*m/v*).

D-2 PROCEDURE

D-2.1 Weigh accurately 4 to 5 g of alkyl benzene sulphonic acid sample. Dissolve in 20 ml of ethyl alcohol. Add a few drops of phenolphthalein and neutralize with sodium hydroxide solution. Add 80 ml of water to the neutralized solution and transfer to a 500 ml separating funnel. Acidify till congo red paper turns blue with dilute hydrochloric acid. Add 15 ml of *p*-toluidine hydrochloride solution. Adjust the *p*H to less than 2 and add 75 ml of ethyl ether. Shake thoroughly with frequent venting and allow the layers to separate. Transfer the acqueous layers to another separating funnel and again add 10 ml of *p*-toluidine hydrochloride solution and extract with 75 ml of ethyl ether. Take down the aqueous layer in 500 ml beaker. Wash the ether layer again with 40 ml of water and drain the aqueous layer in the 500-ml beaker. Discard ether layer.

D-2.2 Warm the aqueous solution in the beaker to expel ether and add 25 ml of barium chloride solution gradually under constant stirring. Digest the precipitate for 1 to 1½ hour. Filter through a previously weighed Whatman No. 1 filter paper or equivalent. Wash the precipitate free from chloride and dry the filter paper to constant mass.

D-3 CALCULATION

D-3.1 Calculate the percentage of free sulphuric acid as follows:

$$\text{Sulphuric acid, percent by mass} = \frac{M_1 \times 41{\cdot}99}{M_2}$$

where

M_1 = mass in g of precipitate, f, and

M_2 = mass in g of sample taken for the test.

ANNEX E

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF COLOUR BY LOVIBOND TINTOMETER

E-0 GENERAL

E-0.1 This method determines the colour of acid slurry by comparison with Lovibond glasses of known colour characteristics. The colour is expressed as the sum total of the yellow and red slides used to match the colour in a cell of specified size in the Lovibond Tintometer.

E-1 APPARATUS

E-1.1 Lovibond Tintometer

E-1.2 Glass Cell — $\frac{1}{4}$ in size.

E-1.3 Filter Paper

E-2 REAGENT

E-2.1 Ethyl Alcohol — 95 percent.

E-2.2 Carbon Tetrachloride

E-3 PROCEDURE

E-3.1 Filter the sample to remove any suspended or extaneous matter. Make sure that the sample in absolutely clear and free from turbidity. Clean the glass cell with carbon tetrachloride and allow to dry.

E-3.2 Prepare a 10 percent solution of acid slurry in 95 percent ethyl alcohol (mass by mass). Fill the glass cell with this 10 percent solution of the sample and place the cell in position in the Tintometer. Place along side of it such red, yellow, blue or neutral Lovibond glass slides or any combination of these as are necessary to match the colour shade of the sample observing colours of the sample and of the combination of the glass slides through an eye piece.

E-4 REPORT

E-4.1 Report the colour of the 10 percent solution of the sample of acid slurry in terms of Lovibond units expressed as yellow and red units.

ANNEX F

[*Table* 1, *Sl No.* (iv) (b)]

DETERMINATION OF COLOUR BY KLETT COLORIMETER

F-1 APPARATUS

F-1.1 Klett Colorimeter

F-2 PROCEDURE

F-2.1 Dilute the acid slurry with distilled water to give a solution containing five percent active matter. Filter the solution in a 4-cm cell using a Whatman No. 42 filter, match the colour of the solution with the standard colour slides. Report as the Klett colour, the number of the matched slide.

ANNEX G

(Foreword)

COMMITTEE COMPOSITION

Soaps and Other Surface Active Agents Sectional Committee, CHD 025

Chairman	*Representing*
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Raw Materials Subcommittee, CHD 025 : 03

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TO
IS 8401 : 1994 ALKYL BENZENE SULPHONIC ACID (ACID SLURRY) — SPECIFICATION

(Second Revision)

(*Page* 1, *clause* **6.2**) — Insert the following at the end:

'**6.2.1** *BIS Certification Mark*

The packages may also be marked with the Standard Mark.

6.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.'

(*Page* 2, *Table* 1, *Note* 2) — Substitute the following for the existing:

'2 For the purpose of calculation, the molecular mass of alkyl benzene sulphonic acid shall be taken as 320.'

(CHD 25)

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